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Solid State Acidic Catalyzed Synthesis of 2-Phenylbenzoxazole

Tổng hợp 2-phenylbenzoxazole sử dụng xúc tác acid pha rắn

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Abstract

Benzoxazole, important to pharmaceutical advancements, was synthesized through the condensation reaction of benzaldehyde and 2-aminophenol under acidic conditions and being characterized using ^{1}H-NMR spectroscopy. This project revealed an efficient method for 2-phenylbenzoxazole synthesis using various acidic catalytic system. A solid catalyst mixture of p-toluenesulfonic acid and silica gel (TsOH-SiO_2) in xylene solvent at 140°C generated the product at 69.66% yield.

Keywords: benzoxazole, solid state catalysis, acidic catalyst

Tóm tắt

Benzoxazole, vốn đóng vai trò thiết yếu trong sự phát triển các sản phẩm dược phẩm, đã được tổng hợp thông qua phản ứng ngưng tụ giữa benzaldehyde và 2-aminophenol trong môi trường acid. Nghiên cứu trong bài báo này giới thiệu một quy trình hiệu quả nhằm điều chế 2-phenylbenzoxazole bằng cách sử dụng các hệ xúc tác rắn acid. Cụ thể, hỗn hợp xúc tác rắn gồm acid p-toluensulfonic và silica gel (TsOH-SiO_2) trong dung môi xylen tại nhiệt độ 140°C đã mang lại sản phẩm benzoxazole với hiệu suất đạt 69,66%.

Từ khóa: benzoxazole, xúc tác thể rắn, xúc tác acid

1. Introduction

Benzoxazole, one of the most important parts of heterocyclic compounds, can be formed by fusing the oxazole ring with a benzene ring. This particular structure exhibits several beneficial biological effects including anticancer, antihypertensive, antiepileptic, antimicrobial, antiviral, antiproliferation, etc. [1-10]. Benzoxazole derivatives, particularly 2-arylbenzoxazole, share similarities with cyclic nucleotides, facilitating their interaction with biological polymers. These compounds are commonly used to create larger derivatives that exhibit further biological activities. Notably, the 2-

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position in the heterocyclic ring holds significant promise for antibacterial effects [11-16].

Synthetic methods for benzoxazole and its derivatives can be categorized into metal-catalyzed cross-coupling and condensation under acidic conditions. Although the metal-catalyzed cross-coupling reactions can generate benzoxazole at high efficiency [17-19], they suffer from many drawbacks such as multiple preparatory steps, harmful solvents, and tedious work-up procedures [20-22]. In contrast, benzoxazoles and their derivatives can be synthesized with less toxicity while maintaining a high yield via a condensation pathway in which strong acids act as catalysts [12, 23-29]. 2-phenylbenzoxaole was prepared with 90% yield by using benzoic acid and o-aminophenol as starting materials in polyphosphoric acid [30]. Moreover, the development of catalytic system of H_2SO_4-SiO_2 possessed several positive characteristics, which were friendly environment, economical and efficient compounds [31-34]. It is, hence, required to develop an acidic catalytic system with a simple procedure, less toxicity, good recovery, and recyclability.

The catalytic system of acids absorbed on the silica gel has gained increasing interests as these systems can offer various benefits. This silica gel- supported approach effectively reduced environmental pollution by minimizing unnecessary waste in various organic conversions. Due to its high thermal stability, large surface area, convenient catalyst retrieval, and effective reaction capacity at typical temperatures, both the catalyst and the method garnered increased attention [35-40].

This study reports the synthesis of 2-phenylbenzoxazole in the presence of different catalytic systems prepared by combining silica gel with various acids including sulfuric acid (H_2SO_4), trifluoroacetic acid (TFA), p-toluene sulfonic acid (PTSA or TsOH), and sodium hydrogen sulfate ($NaHSO_4$) (Figure 1). We envisioned that silica can assist in two main mechanisms: firstly, it helps acids disperse well on the silica gel surface, leading to an increase in the number of effective acid sites, thus increasing the reactant's surface area and its ability to contact the acid. Secondly, silica gel has the ability to adsorb water, one of the reaction products, helping to shift the reaction towards producing more products [41].

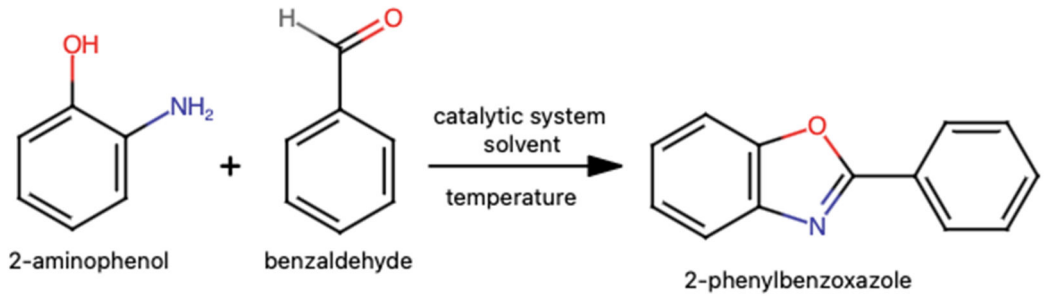

Figure 1. The synthesis of 2-phenylbenzoxazole

2. Material and methods

There were four distinct acidic substances to produce four solid catalysts, including three of acid and one salt, sulfuric acid, trifluoroacetic acid, p-Toluenesulfonic acid and sodium hydrogen sulfate, respectively.

2.1. Preparation of H_2SO_4-SiO_2 catalytic system

A mixture of SiO_2 (50 mmol, 2.95g) and EtOAc (7 mL) in 100 mL flask, was placed in the fridge to cool down the mixture before adding strong acid. 0.5 mL (10 mmol) of H_2SO_4 was then gently added into the flask. The

mixture was stirred for about 1 hour at room temperature to be dissolved completely. The temperature was increased to 77°C for the evaporation of EtOAc. The solid was completely dried to form a free-flowing powder by being heated at 100°C in the oven for about 5 hours.

2.2. Preparation of TFA- SiO_2 catalytic system

SiO_2 (50 mmol, 2.95 g) was suspended in 7 ml EtOAc. The mixture was cooled before addition of TFA (6.5 mmol, 0.5 ml). The mixture was stirred for 1 hour at room temperature. After the mixture was completely dissolved, the temperature was increased to 77°C for the evaporation of EtOAc to obtain the completely dried solid. The achieved powder was heated at 100°C in the oven for about 5 hours.

2.3. Preparation of TsOH-SiO_2 catalytic system

SiO_2 (50 mmol, 2.95 g) was suspended in 7 ml EtOAc. The mixture was cooled before addition of TsOH (10 mmol, 1.7168 g). The mixture was stirred for 1 hour at room temperature. After the mixture was completely dissolved, the temperature was increased to 77°C for the evaporation of EtOAc to obtain the completely dried solid. The achieved powder was heated at 100°C in the oven for about 5 hours.

2.4. Preparation of $NaHSO_4$-SiO_2 catalytic system

SiO_2 (50 mmol, 2.95 g) was suspended in 7 ml EtOAc. Then, $NaHSO_4$ (10 mmol, 1.2 g) was gently added. The mixture was stirred for 1 hour at room temperature. After the mixture was completely dissolved, the temperature was increased to 77°C for the evaporation of EtOAc to obtain the completely dried solid. The achieved powder was heated at 100°C in the oven for about 5 hours.

2.5. Procedure for the synthesis of 2-phenylbenzoxazole

2-aminophenol (87.2 mg, 0.8 mmol) and benzaldehyde (82 μL, 0.8 mmol) were added into a glass vial containing TsOH-SiO_2 catalyst (33.4 mg, 0.2 mmol) and xylene (1.5 mL). The reaction was kept stirring at 140°C. The reaction was monitored by TLC, using Hexane/EtOAc (8:2) as eluent. After the completion of the reaction, the mixture was filtrated to obtain the solution and the catalyst was collected. The product was purified by column chromatography using Hexane/DCM. The solvent was removed by a rotary evaporator.

The ^{1}H NMR of 2-phenylbenzoxazole (Figure 2): ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 – 8.24 (m, 2H), 7.84 – 7.75 (m, 1H), 7.62 – 7.56 (m, 1H), 7.57 – 7.48 (m, 3H), 7.42 – 7.32 (m, 2H).

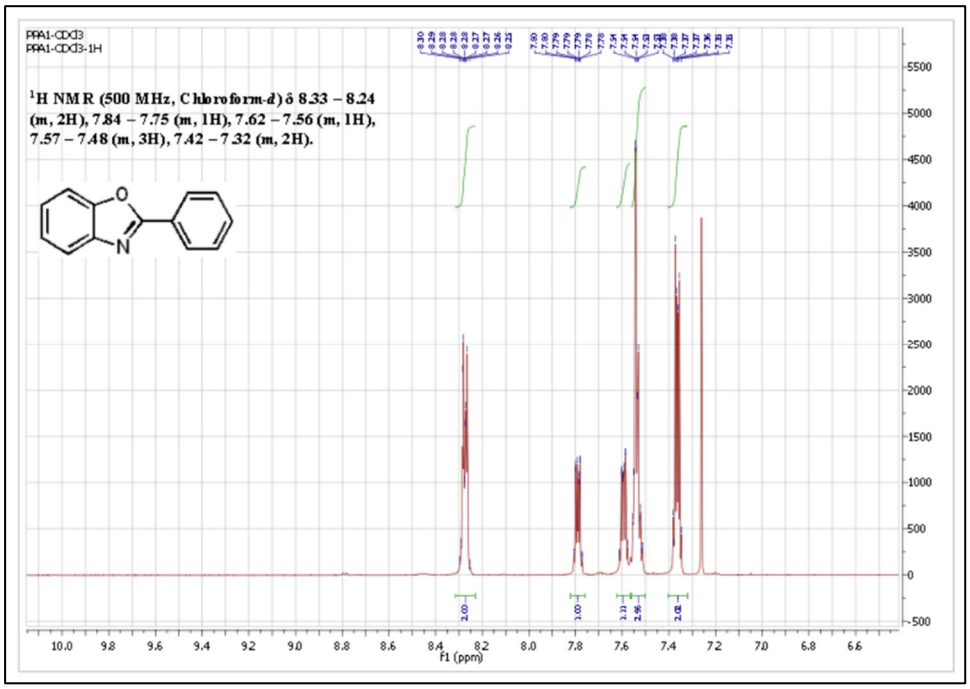

Figure 2. ^{1}H-NMR of 2-phenylbenzoxazole

3. Results and discussion

Six catalytic systems had been used including PPA, TsOH, TsOH-SiO_2, $NaHSO_4$- SiO_2, H_2SO_4- SiO_2, and TFA- SiO_2. Table 1 summarizes the efficiency of each set of conditions in the synthesis of 2-phenylbenzoxazole as in the procedure above. After reaction, the product was purified via column chromatography using Hexane/DCM (5:5) as the eluent and identified by ^{1}H NMR which is consistent with that reported in the literature.

Table 1. The result of each reaction condition for the synthesis of 2-phenylbenzoxazole

Entry	2-aminophenol (eq)	Benzaldehyde (eq)	Benzoic acid (eq)	Catalytic system	Solvent	Temperature (ºC)	Yield (%)
1	1		1	PPA	-	140	23.59
2	1		1	TsOH	Xylene	140	23
3	1	1		TsOH	Xylene	140	37.84
4	1	1		TsOH-SiO_2	Xylene	140	69.66
5	1	1		$NaHSO_4$-SiO_2	Xylene	140	15.37
6	1	1		TFA-SiO_2	Xylene	140	-
7	1	1		H_2SO_4-SiO_2 (19%)	Xylene	140	64.25
8	1	1		H_2SO_4-SiO_2 (38%)	Xylene	140	21.70
9	2	1		TsOH-SiO_2	Xylene	140	20.11
10	3	1		TsOH-SiO_2	Xylene	140	14.79

Xylene was selected as the solvent for this reaction due to its inert nature; thus, ensuring it would not interact with any substances involved, including starting materials, intermediates, or final products throughout the reaction. Various temperatures, including room temperature, 80°C, 90°C, 100°C, 120°C, 140°C, were explored in the process of synthesizing 2-phenylbenzoxazole. Generally, elevating the temperature expedited the reaction by increasing particle collisions. Given the high boiling points of the starting materials — benzaldehyde at 178.1°C and 2-aminophenol at 214°C — there was no obstacle to overcome in the process. Hence, based on the boiling point of o-xylene which is 144°C, the synthesis of 2-phenylbenzoxazole was conducted at 140°C.

Two starting materials that can react with 2-aminophenol to prepare 2-phenylbenzoxazole, benzaldehyde, and benzoic acid, were investigated. The reaction of 2-aminophenol with benzaldehyde exhibited markedly higher efficiency compared to the reaction with benzoic acid (Table 1 – Entry 2 and 3). While the reaction between 2-aminophenol and benzoic acid generated 2-phenylbenzoxazole with 23% efficiency, the condensation of 2-aminophenol and benzaldehyde resulted in a higher yield of 2-phenylbenzoxazole at 37.84%, both reactions underwent similar set of conditions in which TsOH was involved. The less efficiency of benzoic acid could be due to the amide formation more difficult than the imine under the acidic condition. Hence, the remaining samples utilized benzaldehyde and 2-aminophenol as the starting material to prepare 2-phenylbenzoxazole.

The highest yield (69.66%) was achieved through the reactions between 2-aminophenol and benzaldehyde in a 1:1 ratio, facilitated by 0.2 mmol of TsOH-SiO_2 catalyst xylene, conducted at 140°C for 24 hours. Although entry 3 and 4 – Table 1 used the same acid, entry 3 witnessed a considerably lower yield. This explains the effectiveness of SiO_2 involvement in this reaction, which led to less preparatory steps, easy work-up stage, and even distribution of catalyst. Different acidic catalytic systems were prepared, namely, H_2SO_4-SiO_2 with 3 concentrations, TFA-SiO_2, TsOH-SiO_2 and $NaHSO_4$-SiO_2. These catalysts possessed strong acidic characteristics to strongly accelerate the reaction rate. As mentioned previously, in the synthesis of benzoxazoles and its derivatives via condensation, H^+ from the acidic environment was necessary for the preparation step prior to the oxidative cyclization. Thus, the stronger acidity of the catalyst could support the synthesis generally and effectively. The order of acidity among these acids, determined by their pKa values, goes as follows: H_2SO_4 (-3.0), TsOH (-2.8), TFA (0.52), and $NaHSO_4$ (1.99). Since the acid with smaller pKa value is a better source of protons for the reaction, the H_2SO_4-SiO_2 was predicted to be the most effective catalyst. Regarding H_2SO_4-SiO_2, three different concentrations — 3%, 19%, and 38%—were examined. The 3% catalyst proved ineffective due to its limited acid content since the higher concentrations of acid in the catalyst was expected to enhance the catalyst's reactivity in accelerating the reaction. However, the reaction utilizing 38% H_2SO_4-SiO_2 was only 21.70% efficiency. The excessive density of acid in the catalytic mixture can be used to explain for this phenomenon as it might interact with other substances and thereby generate by-products, resulting in a lower yield (Entry 7 – Table 1). The 19% H_2SO_4-SiO_2 emerged as the most compatible catalyst (Entry 6 – Table 1) with 64.25% efficiency. Based on the results achieved, the sample using TsOH-SiO_2 catalyst exhibited the highest efficiency at 69.66%. This happened because the sulfate group in H_2SO_4 tended to reactively interact with other

substances, causing the formation of by-products, whereas TsOH catalyzed the mechanism without undergoing reactions with others, enabling a more seamless progression. Additionally, as entries 9, 10 – Table 1 illustrate, an increase in 2-aminophenol was associated with a lower reaction efficiency. Higher reactant concentration generally results in higher particle density, which in turn accelerates the reaction rate by causing more collisions to produce products. However, the 2-aminophenol may interact with other substances in the mixture generating by-products which resulted in difficulties in the purification steps and lower yield of desired product.

4. Conclusion

To conclude, this study investigated an effective route for synthesizing 2-phenylbenzoxazole through a condensation reaction involving benzaldehyde and 2-aminophenol. Different acidic catalysts were prepared and utilized aiming to enhance the reaction efficiency with convenient preparation steps, easy work-up stage, and the usability of catalyst. A yield of 69.66% was attained by the reaction of 2-aminophenol and benzaldehyde in equal proportions, aided by 0.2 mmol of TsOH-SiO_2 catalyst in xylene, performed at 140°C for 24 hours.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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